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ENGINEER RESEARCH AND DEVELOPMENT LABORATORIES

Report 1102

THE PLASTIC DEFORMATION OF THALLIUM HALIDES IN RELATION TO CRYSTAL ORIENTATION

Project 8-23-02-002

14 January 1949

Submitted to

THE CHIEF OF ENGINEERS, U. S. Army

by

The Commanding Officer
Engineer Research and Development Laboratories

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FOREWORD

The material here presented is the result of work carried on by Dr. Alexander Smakula and Myron W. Klein of the Radiation Branch, Engineer Research and Development Laboratories, Fort Belvoir, Virginia, during 1948. A brief resume of this report was presented at a meeting of the Optical Society of America, on 23 October 1948, at Detroit, Michigan.

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ABSTRACT

The plastic deformations of cubic thallium halide crystals, produced under pressure of a cone perpendicular to the crystal surface, are seen as two kinds of surface patterns, the superficial and the transmitted, with characteristics dependent on the crystal orientation. Superficial patterns have wings radiating from the press point; transmitted patterns are square or rhombic. The number, orientation, shape, and length of the wings of the superficial patterns, and the shape and position of the transmitted patterns, depend on the orientation of the crystal with respect to the surface of the crystal sample. Characteristics of the patterns are explained by the mechanism of plastic deformation in crystals. Application of the punch patterns to determination of crystal orientation preparatory to processing the crystals into optical elements is explained.

THE PLASTIC DEFORMATION OF THALLIUM HALIDES IN RELATION TO CRYSTAL ORIENTATION

I. INTRODUCTION

1. Subject. This report covers an investigation of the plastic deformation of cubic crystals of thallium halides in relation to crystal orientation. The method employed may be termed "punch-pattern method," in which a sharp cone is pressed into the crystal and the resulting surface deformation patterns and internal strains are studied.

2. Authority. The study was conducted under authority of a letter from the Chief of Engineers to the Engineer Board (Engineer Research and Development Laboratories), dated 14 October 1946, file ENGNC (14 Oct 46), subject: Infrared Optical Materials, Approval of Military Characteristics and Authorization of Development Project XR 750.

3. General. For many years natural and synthetic crystals (such as the alkali halides and silver chloride) have been used in the form of prisms, lenses, and windows for scientific and applied investigations in the infrared. These crystals, however, have properties which make them difficult to employ, particularly in field equipment. The high water solubility of the alkali halides (i.e., NaCl, KBr) is a serious disadvantage because exposure to moist air destroys optical surfaces. The resistance to moisture of optical elements made from crystals of alkali halides may be improved by suitable coatings, but such improvement is not sufficient to permit their use under average humidity conditions. Silver chloride, on the other hand, is stable in the presence of moisture, but is sensitive to light and blackens in the same manner as a photographic plate. In addition, its very low hardness makes grinding and polishing quite difficult.

In the past few years, crystals of thallium halides have been grown and investigated for use as optical elements in the infrared.[1] These crystals have some properties better than those of

1. W. R. Brode and L. E. Mayes, German Infra-Red Devices and Associated Investigations, CIO Subcommittee Report, File No. XXX-108, Items Nos. 1 and 9, 41-43 (1945).
A. Elliot and L. E. Mayes, German Infra-Red Devices and Associated Investigations, CIO Subcommittee Report No. 2, File XXXIII-9,8-10 (1945).
ERDL Report 1076, Production of Infrared Transmitting Thallium Halide Crystals, 8 October 1948.
R. Koops, Optik, 3, 298 (1948).
G. Hettner and G. Leisegang, Optik 3, 305 (1948).

alkali and silver halides. Their water solubility is sufficiently low that exposure to average humidity conditions does not harm optical surfaces. They are practically insensitive to visible light. The hardness of crystals of thallium chloride (TlCl) and thallium bromide (TlBr) is approximately equal to that of silver chloride and this low hardness is a disadvantage. However, the hardness of the crystals of mixed thallium bromide and iodide and of mixed thallium chloride and bromide is approximately three times greater. The crystals of mixed thallium bromide and iodide, known as KRS-5,[2] because of their hardness, high refractive index (2.461 at 1 micron, 2.366 at 16 microns, and 2.291 at 32 microns), and high transmission over a wide range (approximately 70 percent for wavelengths from one to 35 microns with cutoff at 40 microns), are finding important applications.[3]

In order to fabricate and employ optical elements of thallium halide crystals most efficiently, it is necessary to know, in addition to the optical properties which have been determined, such mechanical properties as hardness, elasticity, photoelasticity, and plasticity. Unlike the optical properties, the mechanical properties depend on the orientation of the crystal lattice. The subsequent paragraphs of this report describe an investigation of the plastic deformation of thallium halides in relation to crystal orientation.

II. INVESTIGATION

4. Methods and Equipment Used for the Punch-pattern Investigation. The common method for investigating plastic deformation consists of placing the crystal sample in a stout steel cylinder and then pressing on an exposed surface, as described by Buerger.[4] This method is tedious for series investigation. Therefore, a simpler method, the "punch-pattern method," first employed by Reusch, was used in this study.[5] Tammann and Mueller[6] have used the punch-pattern method for determining the orientation of large metal

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2. KRS-5 is the code name assigned to these crystals by the German developers during World War II.

3. E. K. Plyler, "Prism Spectroscopy from 24 to 37 Microns," Journ Chem Phys, 15, 885 (1947).
R. Hofstader, "Thallium Halide Crystal Counter," Phys Rev, 72, 1120 (1947).

4. M. J. Buerger, Am. Mineral 15, 45 (1930).

5. E. Reusch, Ann. der Phys. und Chem. 132, 443 (1867).

6. J. Tammann and A. Mueller, Zs.f. Metallkunde 18, 69 (1926).

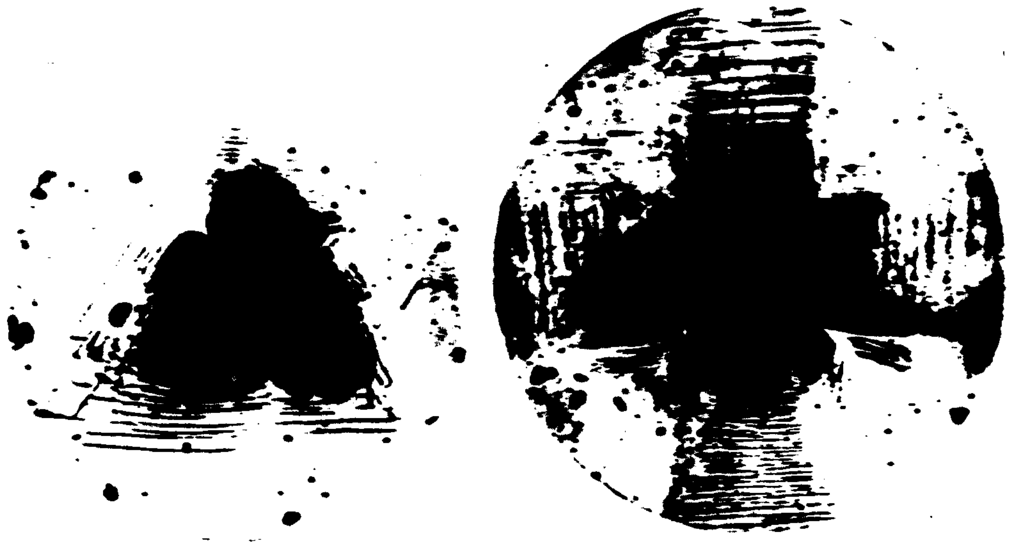

Fig. 1. Press patterns on Cu-crystal (after Tamman and Muller). Left: octahedron face, magnification 52X; right: cube face, magnification 88X.

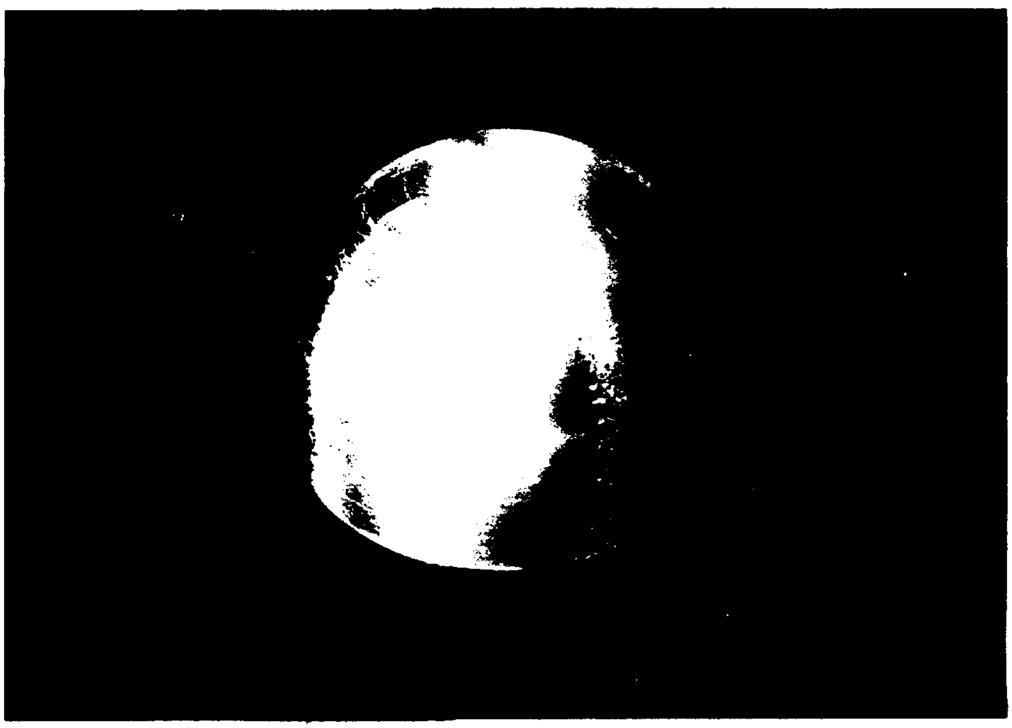

Fig. 2. Crystal hemisphere of KRS-5 before polishing.

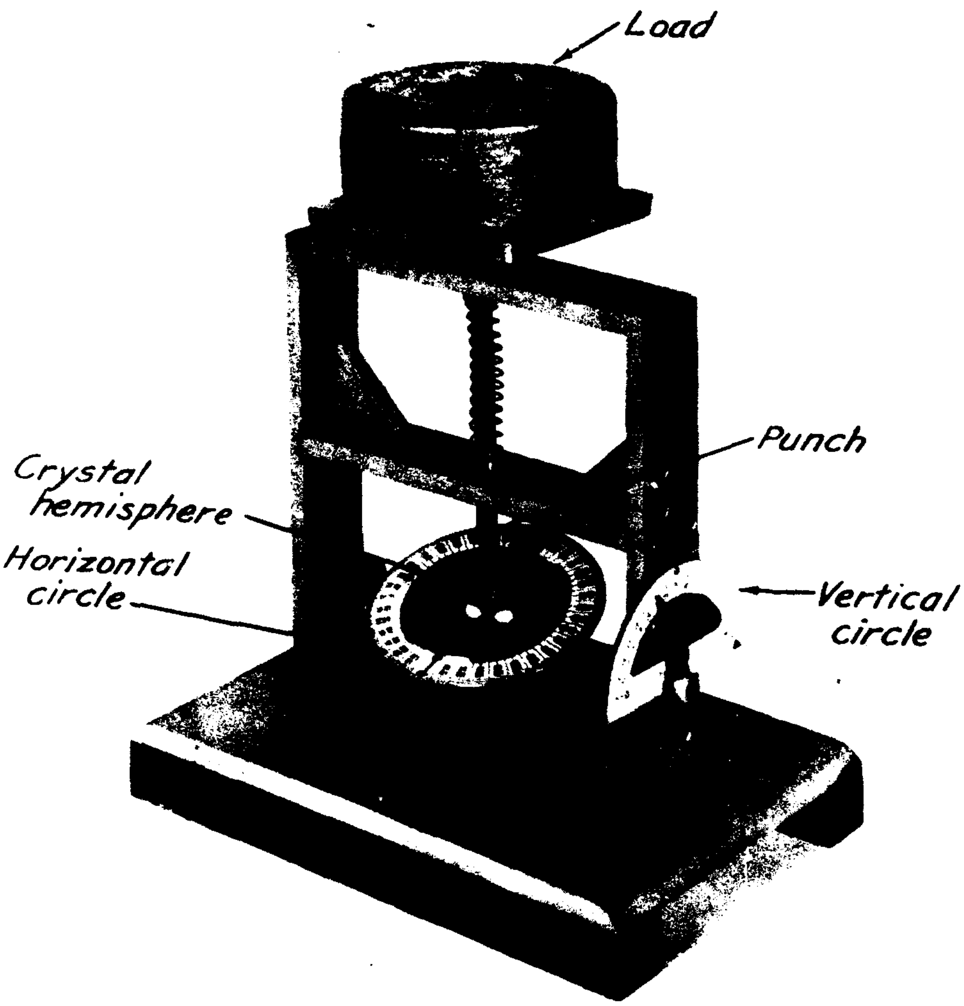

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Fig. 3. Pressing goniometer with horizontal and vertical circles.

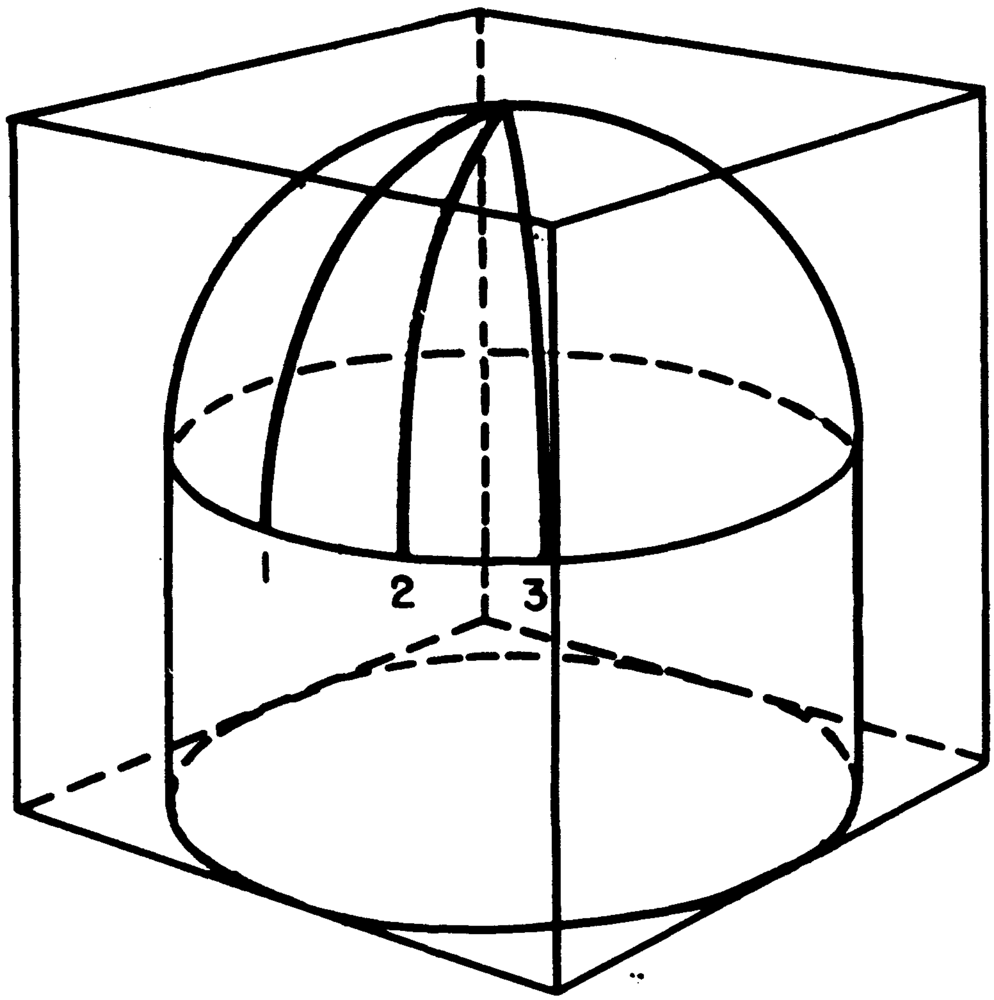

Fig. 4. The model of KRS-5 sphere used for punch patterns. The cube indicates the crystal orientation. The lines marked 1, 2, 3, are the lines along which the three series of press patterns shown in Figs. 6, 8, and 9, were taken. Line 1 runs from cube face to cube face, line 2 makes an angle of $22\frac{1}{2}$ degrees with line 1, and line 3 makes an angle of 45 degrees with line 1.

crystals (Fig. 1). The method consists of pressing a needle into a crystal surface. Around the press point lifted patterns appear which can be seen under a microscope. The shape and symmetry of the patterns depend on crystal orientation. To enlarge the patterns, a cone with an apex angle of 80 degrees was used in this study.

In order to expose all possible crystal orientations, a hemisphere of KRS-5 with a radius of 2.5 cm was prepared with its base parallel to a cube face. The hemisphere, after having been turned to shape on a lathe, is shown in Fig. 2. The rough bands spaced 90 degrees apart around its axis indicate a difference of behavior of various crystal orientations under the cutting tool. The hemisphere was finally polished by optical methods for the punch-pattern investigation.

In order to press the conical punch into any desired crystal orientation, a pressing goniometer (Fig. 3) was constructed with horizontal and vertical circles capable of an accuracy within one degree. Best results were obtained with a load of 20 pounds on the punch. With this load the punch sinks into the surface to a depth of about 0.5 mm and produces a hole of approximately 0.8-mm diameter at the surface.

5. Superficial Punch Patterns of KRS-5. The KRS-5 crystals have a body-centered cubic lattice. When the punch is pressed into the surface, one to four raised wings which may be seen by the unaided eye appear around the hole. The number, length, and symmetry of these wings, which might be called "superficial patterns," depend on orientation of the crystal. According to Tammann and Mueller,[7] the punch pattern on the cubic face (001) should have fourfold symmetry; on the dodecahedron face (110), twofold symmetry; and on the octahedron face (111), threefold symmetry.

Three series of punch patterns were made in angular steps of approximately 10 degrees along the lines shown in Fig. 4. The series on line 1 of Fig. 4 runs from one cube face (001) to another cube face (100). The series on line 3 continues from the cube face (001) through the octahedron face (111) to the dodecahedron face (110). The series on line 2 runs from the cube face to the base of the hemisphere along the bisector of the angle formed by lines 1 and 3. The punch patterns were photographed by the apparatus shown in Fig. 5.

The patterns produced along line 1 of Fig. 4 are shown in Fig. 6. On the cube faces (001) and (100), 0 and 90 degrees, respectively, on Fig. 6, there is a slight indication of fourfold symmetry. Actually, under the proper lighting conditions, there can

7. J. Tammann and A. Mueller, Zs.f. Metallkunde 18, 69 (1926).

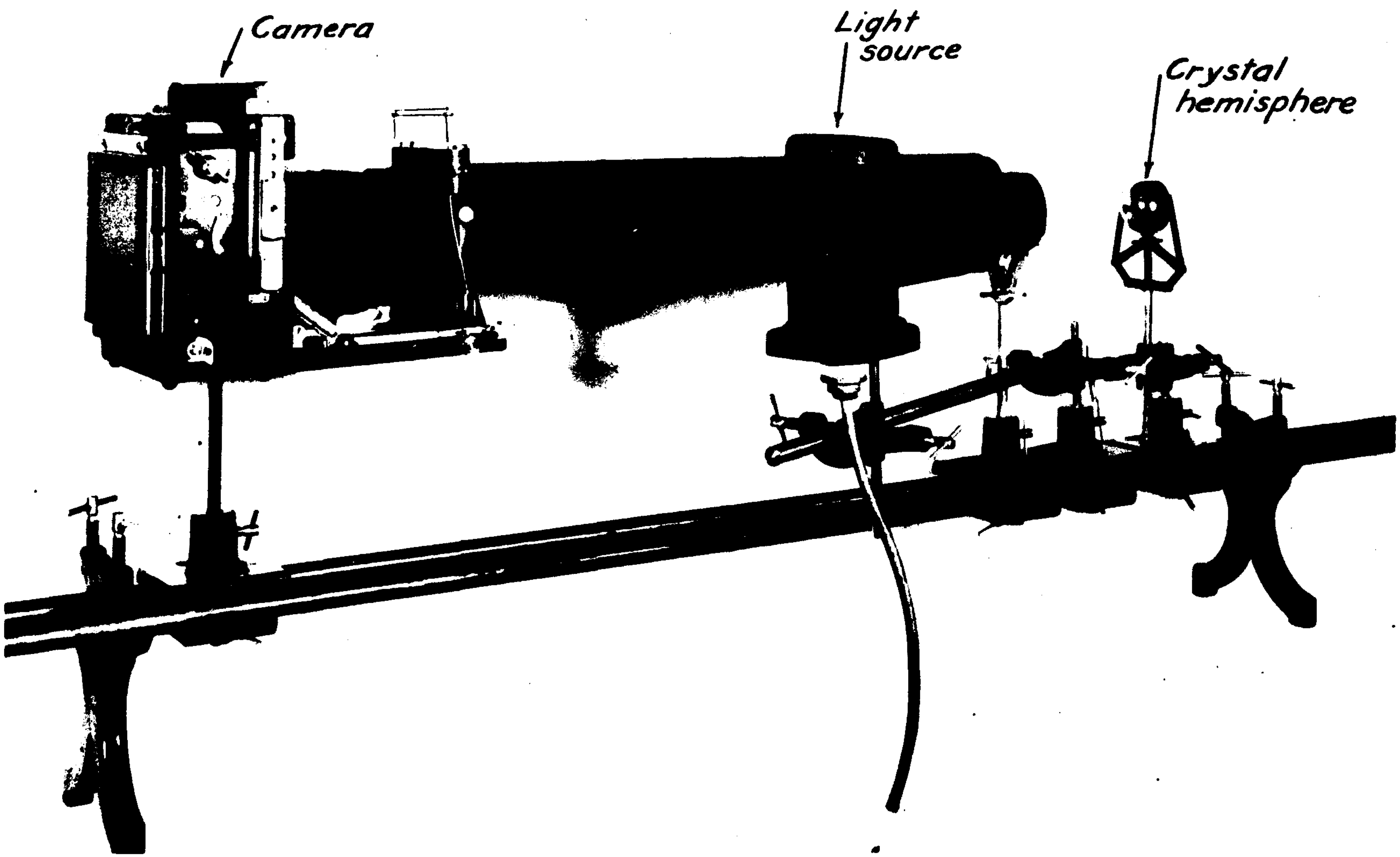

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Fig. 5. Apparatus for taking photographs of the punch patterns.

be detected four long, weak wings running parallel (or perpendicular) to cube edges, as shown in Fig. 7. The wings transverse to the long axis of the paper of Fig. 7, (i.e., transverse to line 1 of Fig. 4) persist for all punch patterns along line 1 of Fig. 4, but show in Fig. 6 only for the photographs at 10 and 80 degrees. As the angle in respect to the cube face is increased, the wing parallel to line 1 of Fig. 4 and opposite to the direction of angular change (upper wing of Fig. 7) disappears within an angular change of two to three degrees. With angular change, the wing on the opposite side of the hole (the wing parallel to line 1 of Fig. 4 and in the same direction as angular change) becomes shorter and stronger. At 45 degrees, both winds, very short and equal in length, are seen. As the angle is further increased, theupper wing (wing opposite to the direction of angular change) becomes longer and weaker and the lower wing (wing in same direction as angular change) disappears.

Fig. 8 shows the sequence of patterns obtained in the direction from the cube face (001) through the octahedron face (111) to the dodecahedron face (110); i.e., along line 3 of Fig. 4. As the angle is increased, the two weak wings on the cube face opposite to angle change, (Fig. 7) quickly disappear and the other two long symmetrical wings become stronger as shown in the 10-degree photograph of Fig. 8. With further increases in angle, these wings become progressively shorter and stronger, while a third wing develops. The angle between the two original wings increases and reaches 120 degrees at the face (111) where all three wings are equal in size with equal angles between them. As the angle is further increased to 90 degrees, the third wing becomes longer and then disappears at the cube edge, while the angle between the two original wings becomes 180 degrees.

Fig. 9 shows the sequence of patterns obtained in progressing from the cube face (001) along line 2 of Fig. 4. The changes of the wings are similar to those of the wings of Figs. 6 and 8, except that symmetry does not exist.

Fig. 10, 11, and 12 illustrate the change of the lengths of the wings shown on Fig. 6, 8, and 9, respectively, as the press point moves progressively around the crystal through 360 degrees.

In progressing from cube face to cube face around the crystal (Fig. 10) each of the two wings shown on Fig. 6 consecutively go from zero length to maximum length and then back to zero length with both wings being present on cube face planes and in the region of cube edges. This sequence is repeated twice. On cube-face planes, the wings are the longest; while, in the region of cube edges, they are the shortest.

In advancing from cube face (001) through octahedron face (111) to dodecahedron face (110) through 360 degrees (Fig. 11),

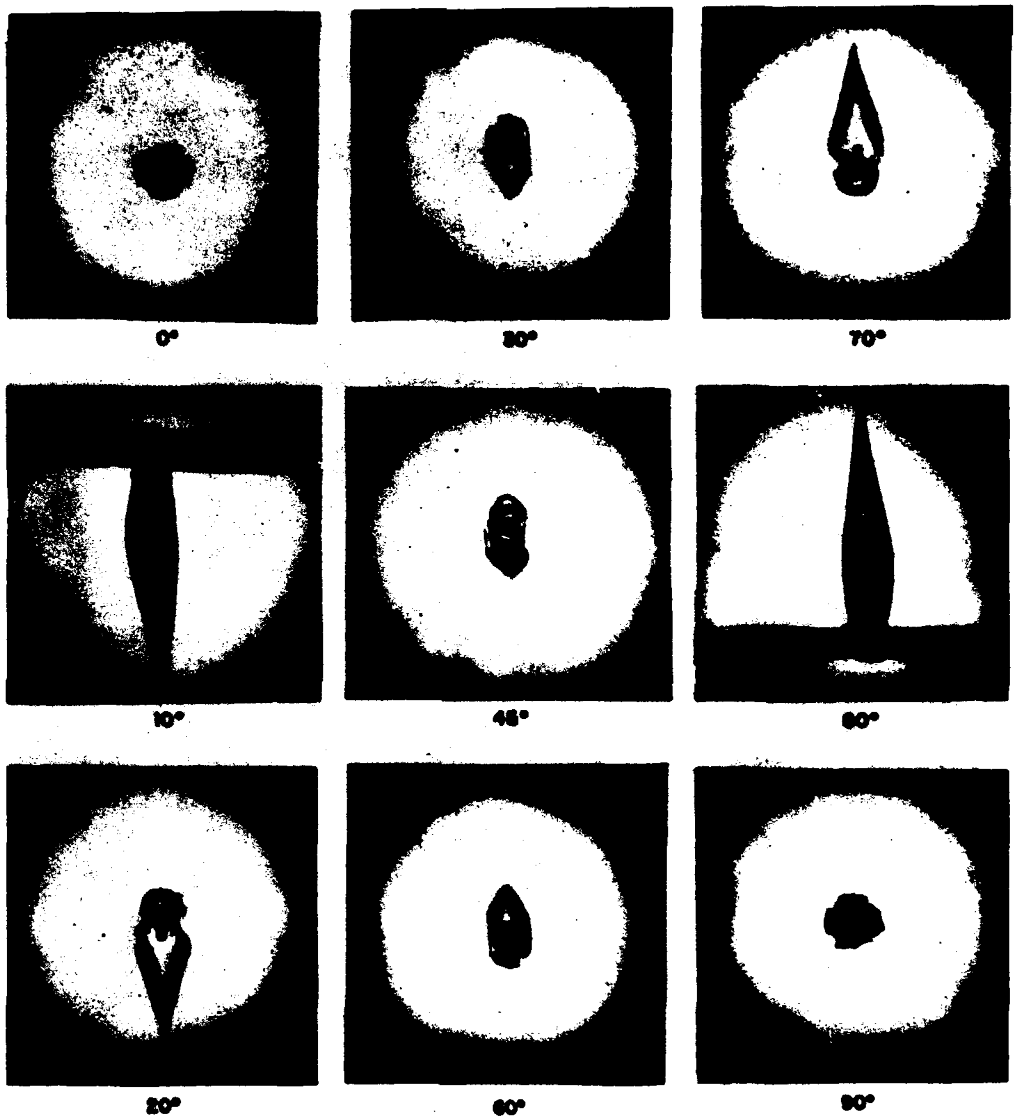

Fig. 6. Punch-pattern sequence from one cube face (001) to the next (100) made along line 1 of Fig. 4. Magnification 12X. At 0 and 90 degrees (cube faces), proper lighting conditions permit detection of the four long, weak wings as shown in Fig. 7. The wings transverse to line 1 of Fig. 4 persist throughout the sequence, but show only for 10 and 80 degrees in the above photographs.

Fig. 7. Long, weak punch wings formed on a cube face.

the length of the pair of wings shown on Fig. 8 becomes shorter and reaches a minimum at 54 degrees on the (111) face. It remains approximately constant for the next 72 degrees and then increases to the maximum at 174 degrees. The third wing on Fig. 8 which starts to develop at approximately 30 degrees, reaches a maximum at 80, 100, 260, and 280 degrees as shown on Fig. 11, but disappears on the dodecahedron face (110), i.e., at 90 and 180 degrees. Both wing patterns consecutively go from a maximum length through a minimum back to maximum around the crystal. This sequence is repeated twice.

Fig. 12 shows the changes in length of the wings shown on Fig. 9 as the press point progresses from a cube face along a path midway between those of Figs. 10 and 11 (or of Figs. 6 and 8). Two of the wings change length simultaneously followed by change of length of the third wing, similarly to the sequence of Fig. 11.

The length of the wings, of course, also depends on the force applied by the press point. In Fig. 13 are shown typical curves of wing length versus load on the punch for two crystal orientations. Both curves approach a maximum value.

6. Strain Patterns of KRS-5. In addition to the superficial patterns, the press point causes strains just below the surface of the crystal. These become visible when the crystal is placed between crossed polaroid screens. Photographs of the strain patterns, made with the apparatus shown in Fig. 14, are given by Figs. 15, 16, and 17. Like the superficial patterns, the strain patterns exhibit characteristic shapes corresponding to the crystal orientation.

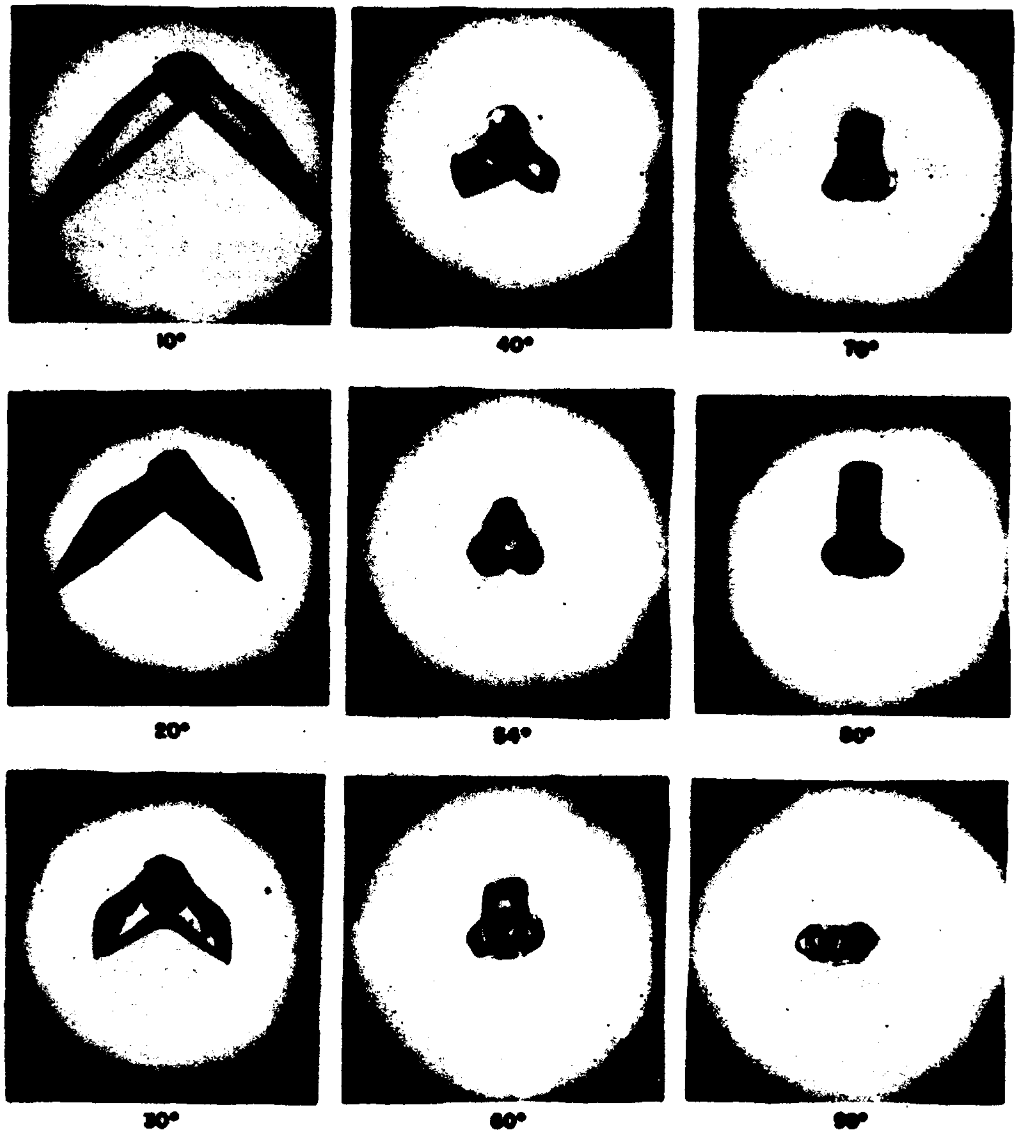

Fig. 8. Punch-pattern sequence from cube face (001) through the octahedron face (111) to the dodecahedron face (110), made along line 3 of Fig. 4. Magnification 12X.

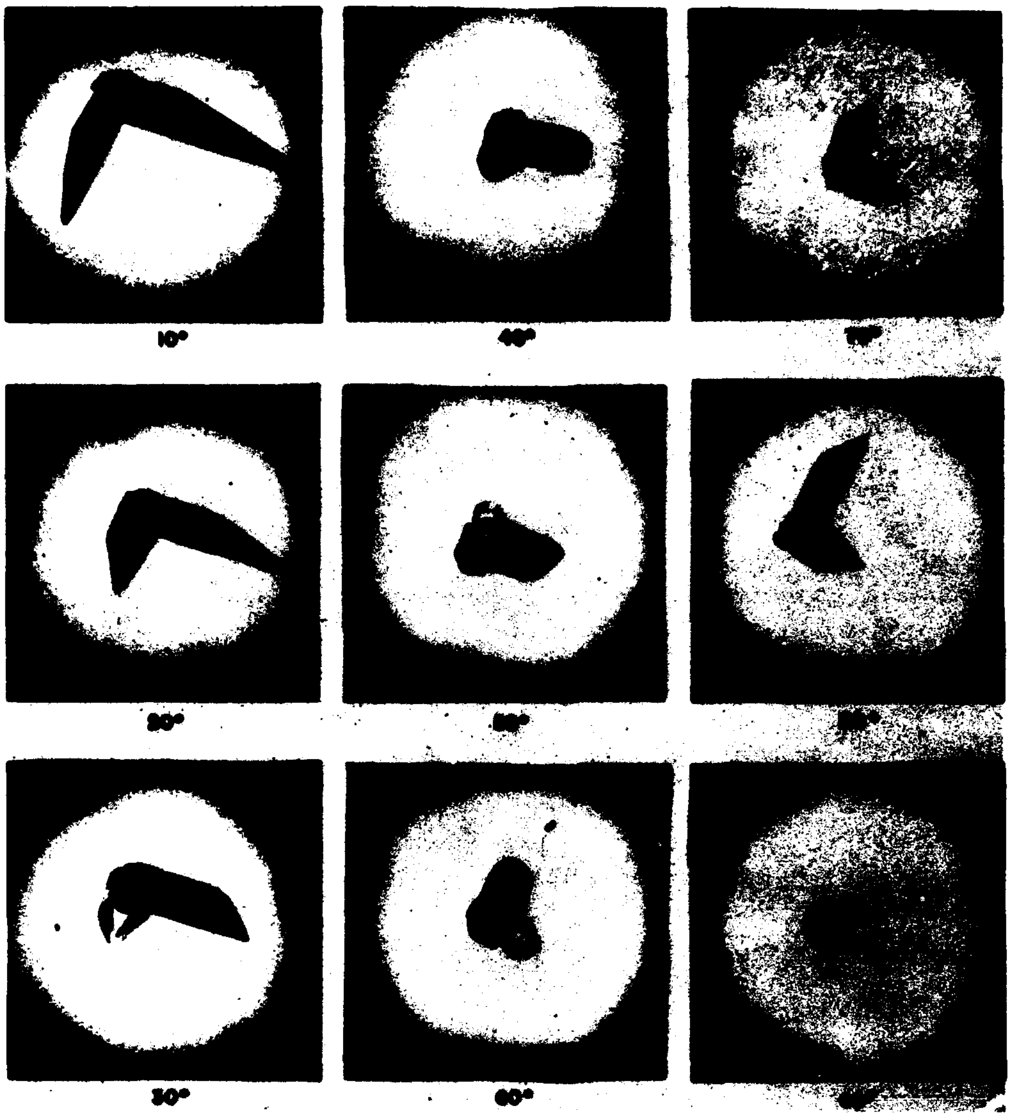

Fig. 9. Punch-pattern sequence from cube face (001) along the path of line 2 of Fig. 4; i.e., the path midway between those of Figs. 6 and 8. Magnification 12X.

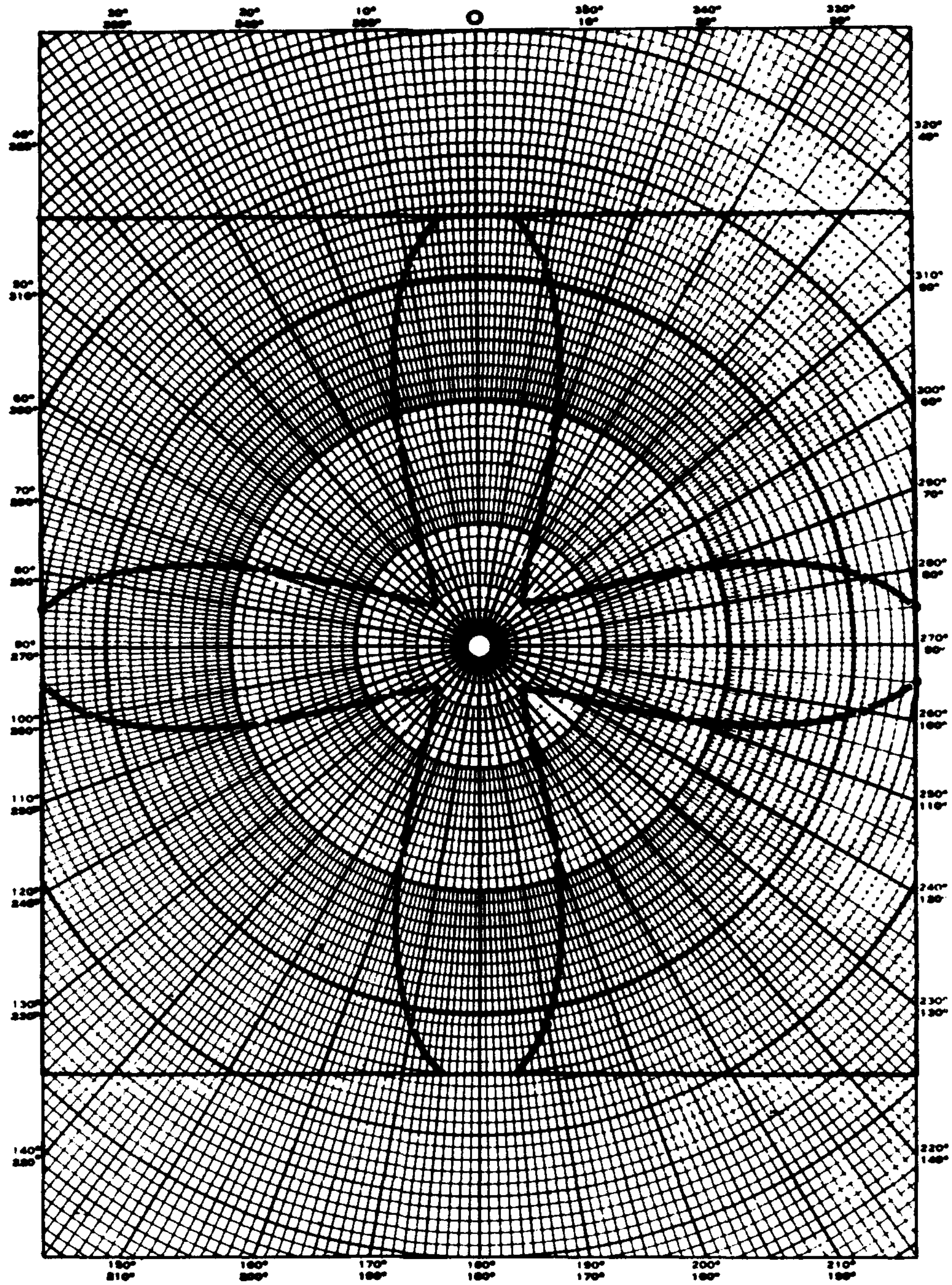

Fig. 10. Change of length of the wings shown in Fig. 6 as the press point moves progressively from cube face to cube face through 360 degrees.

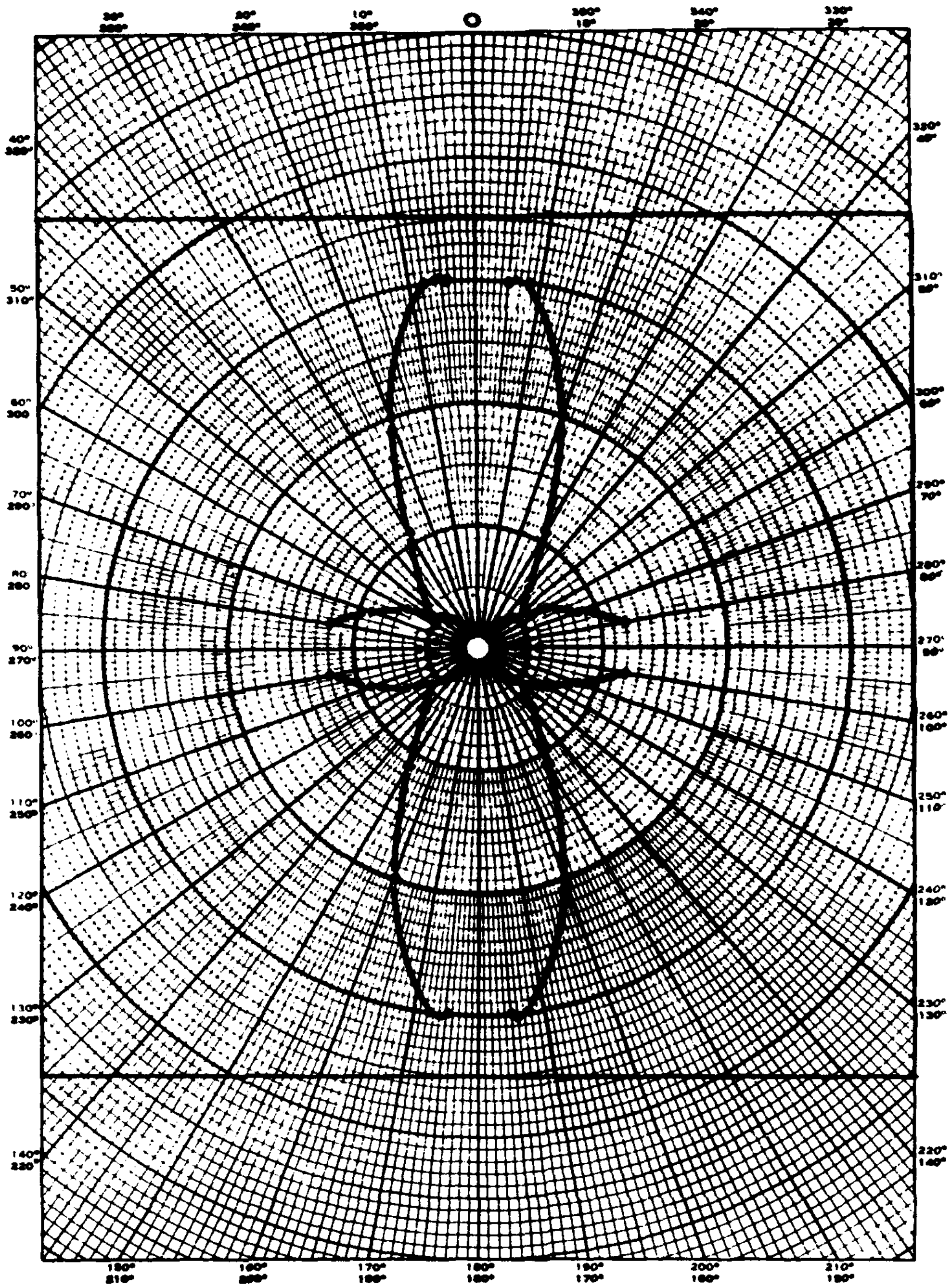

Fig. 11. Change of length of the wings shown in Fig. 8 as the press point moves progressively from cube face through octahedron face to dodecahedron face through 360 degrees.

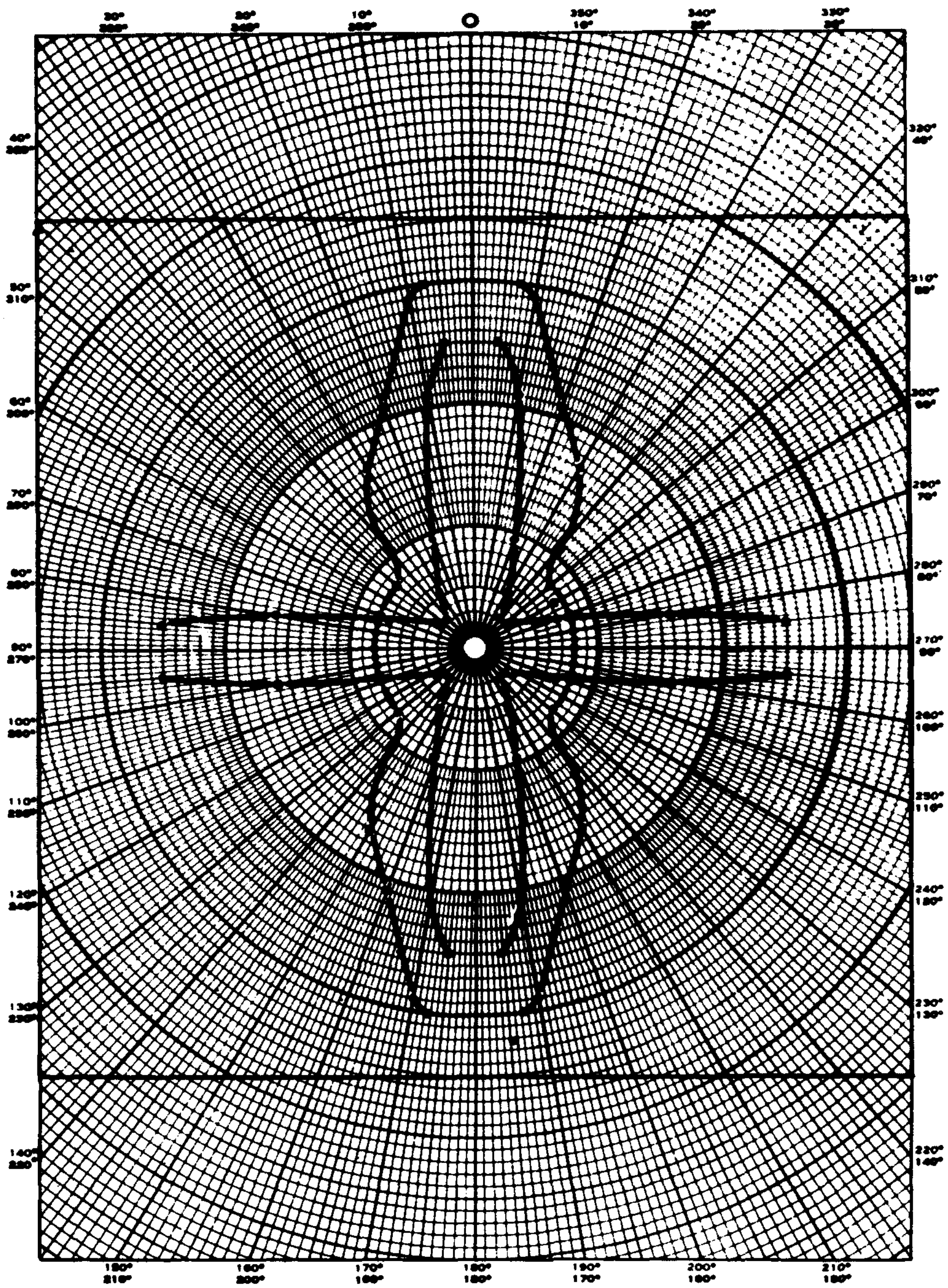

Fig. 12. Change of length of the wings shown in Fig. 9 as the press point moves progressively through 360 degrees along a path midway between those of Figs. 11 and 12 (or of Figs. 6 and 8).

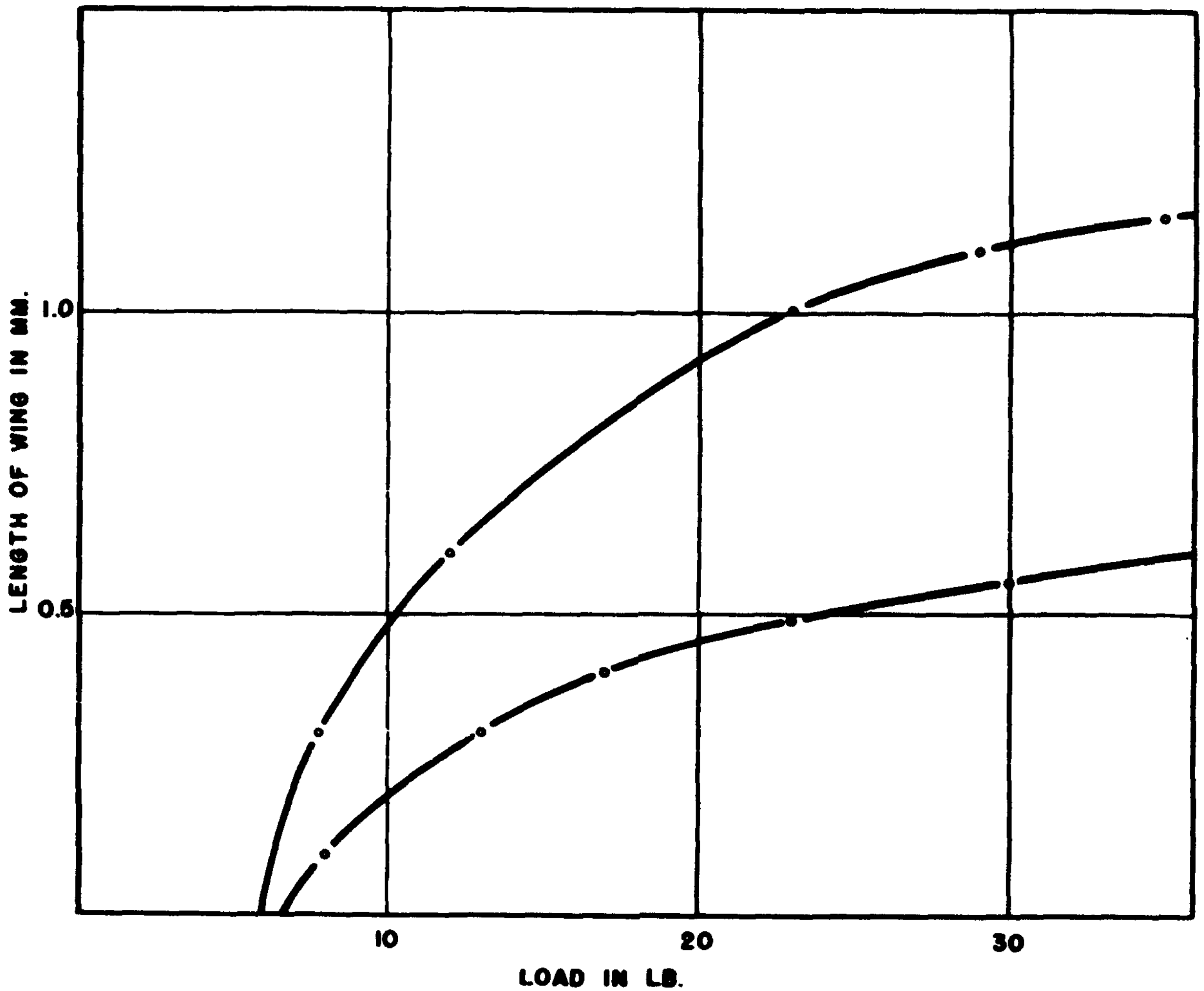

Fig. 13. The influence of the load on the length of the press patterns. Lower curve: 110-face; upper curve: surface between 001- and 110-face.

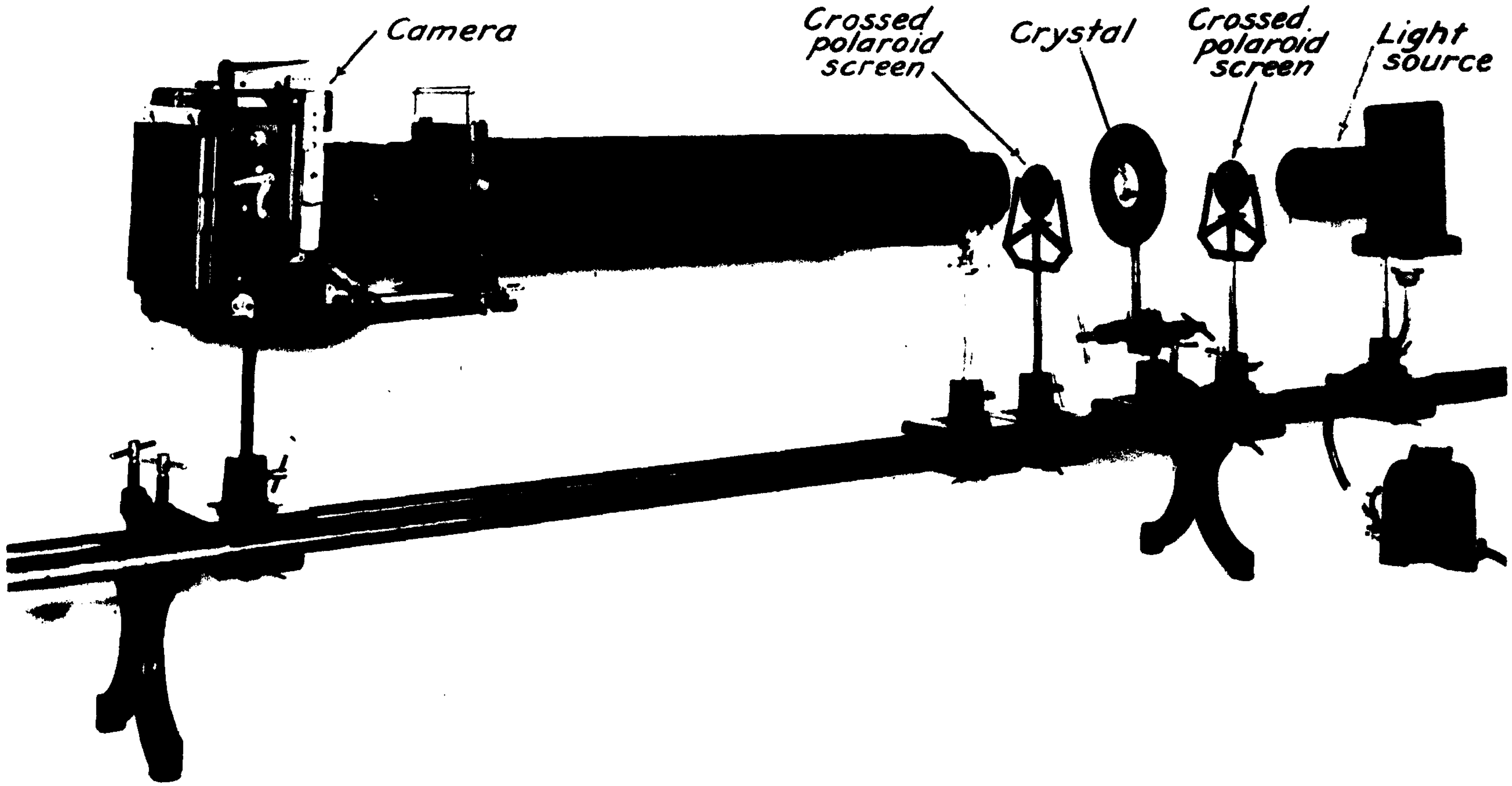

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Fig. 14. Apparatus used for photographing strain patterns.

7. Transmitted Patterns of KRS-5. If crystal samples only a few millimeters thick are used, a "transmitted pattern" such as that shown in Fig. 18 is produced on the underside of the sample. The shape of the transmitted pattern and its position depend on orientation of the crystal. This is illustrated by Figs. 19, 20, and 21, where both superficial and transmitted patterns are shown for three principal crystal orientations. Pressing into a plate with surfaces parallel to the cube face produces a raised square transmitted pattern in the form of a truncated pyramid directly opposite the press point. If either or both surfaces of the crystal sample are not parallel to the cube face, the transmitted pattern becomes somewhat rhombic and is displaced laterally from the position directly opposite to the press point. Pressing on a dodecahedron face produces two transmitted patterns on the opposite cube faces, and pressing on the octahedron face produces three transmitted patterns.

8. Punch-pattern Investigation of Other Crystals. Similar press patterns were produced on other cubic crystals of thallium halides, i.e., thallium chlorobromide,[8] thallium chloride, and thallium bromide (thallium iodide crystallizes rhombic). When the punch-pattern method was applied to silver chloride, instead of sharp wings a diffuse fourfold symmetrical pattern appeared (Fig. 22), which seemed to be independent of crystal orientation. In the case of sodium chloride, pressing on a cube face resulted principally in fourfold splitting roughly along the diagonals of the cube face as illustrated by Fig. 23. When the experiment on sodium chloride was repeated at a temperature of approximately 200 C, the resulting press pattern was very similar to that obtained on silver chloride at room temperature.

III. DISCUSSION

9. Mechanism of Plastic Deformation. The experimental results show the plastic deformations produced in cubic thallium halide crystals by pressure of a cone perpendicular to the crystal surface. The plastic deformations are seen as two kinds of surface patterns caused by material displacement in the crystal: superficial patterns around the press point; and, if the crystal sample is sufficiently thin, transmitted patterns on the opposite side. Superficial patterns have wings radiating from the press point; transmitted patterns are square or rhombic. The number, orientation, shape, and length of the wings of the superficial patterns, and the shape and position of the transmitted patterns, depend on the orientation of the crystal with respect to the surface of the crystal sample.

8. Designated as KRS-6 by the German developers of this crystal.

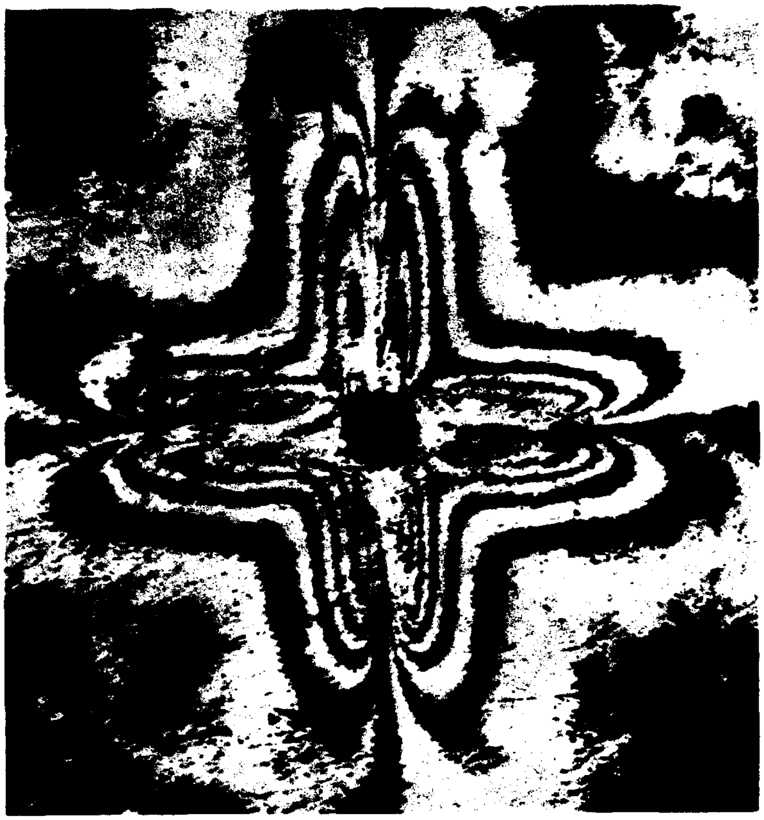


Fig. 15. Inner strains formed by pressing on the 001-face of KRS-5.

Fig. 16. Inner strains formed by pressing on the 110-face of KRS-5.

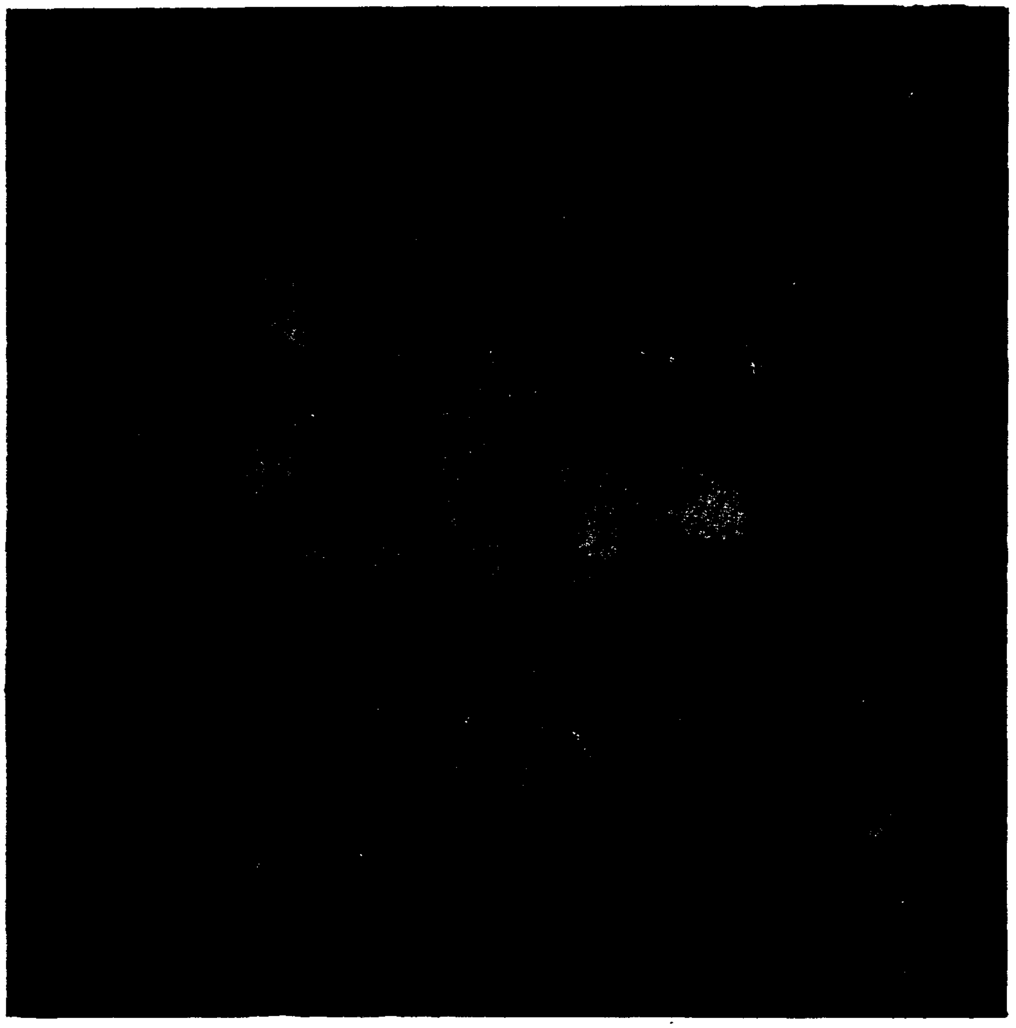
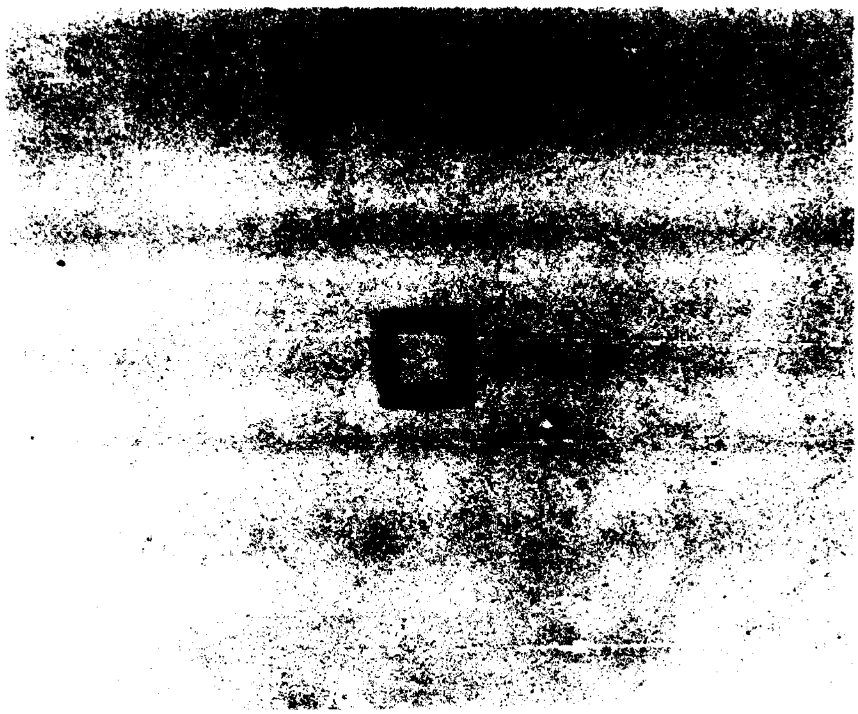


Fig. 17. Inner strains formed by pressing on the 111-face of KRS-5.



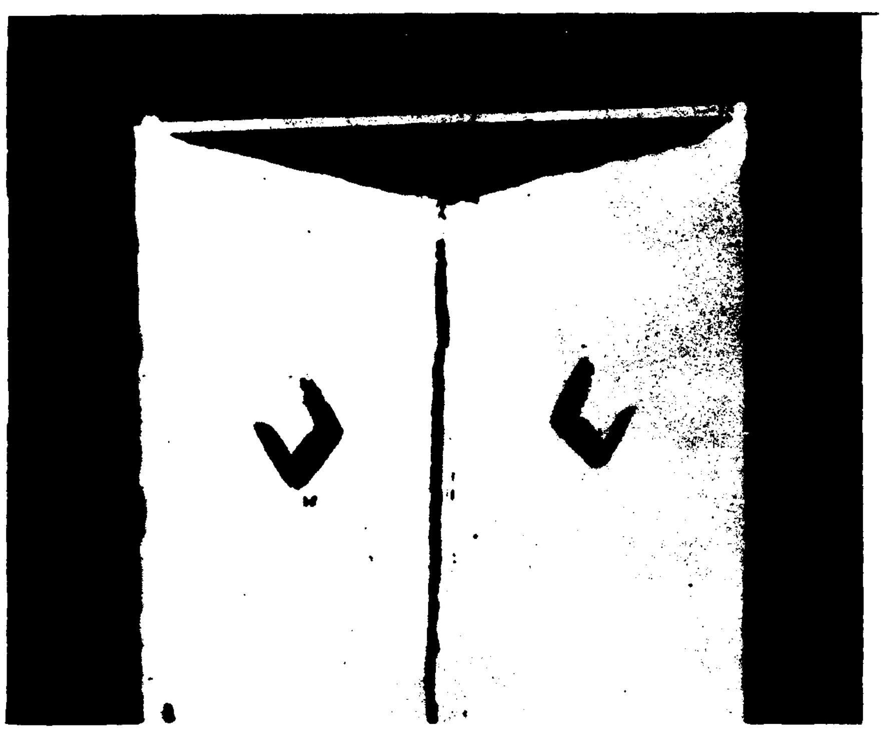


Fig. 18. Transmitted press patterns on KRS-5. Top: by pressing on the cube face; bottom: by pressing on the dodecahedron face.

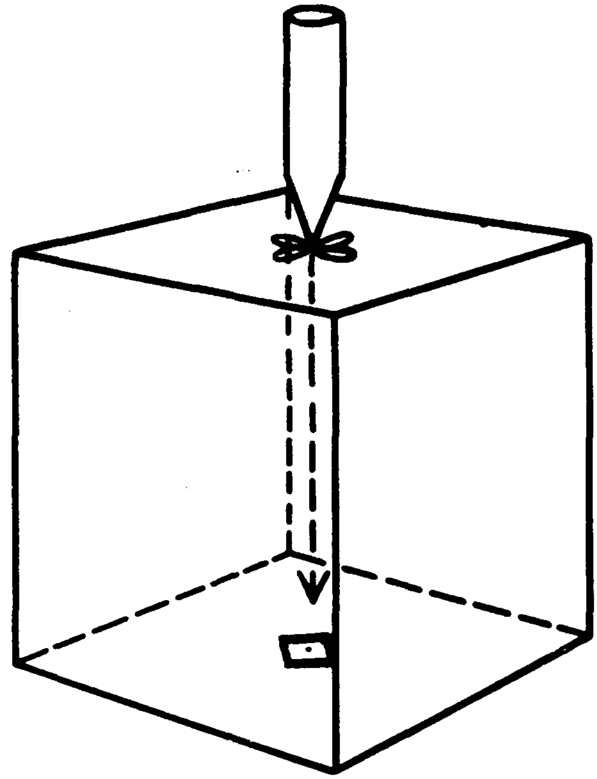


Fig. 19. Superficial and transmitted patterns formed by pressing on a cube face.

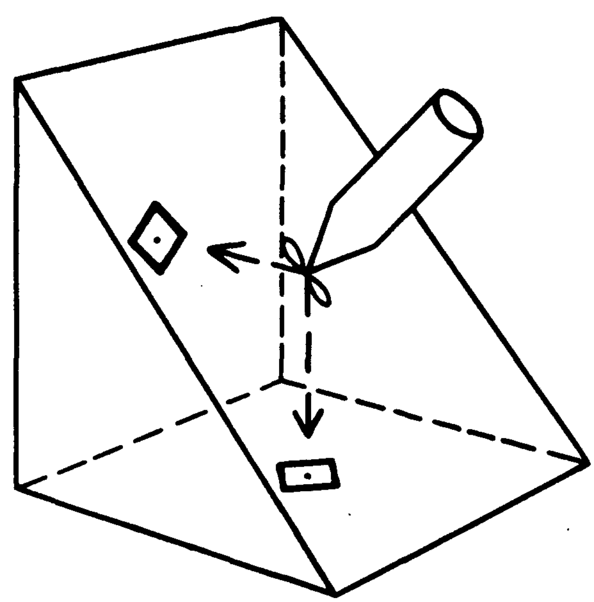


Fig. 20. Superficial and transmitted patterns formed by pressing on a dodecahedron face.

In order to explain the change of the patterns with respect to change in crystal orientation, the mechanism of the plastic deformations must be known. The following explanation of the mechanism of the deformations under unidirectional tension and compression, based on investigations made on alkali halide crystals, has been made.[9] Under the applied pressure, the single crystal is broken into many small parts which slip and rotate along the slip planes in the slip directions. Thus a plastically deformed region is no longer a single crystal but an aggregate of small crystals.

It may be expected that a similar mechanism is responsible for the patterns obtained on KRS-5 and other cubic thallium halide crystals in these investigations, although the pressure was applied in a different manner.

In sodium chloride crystals the slip plain is parallel to the cube face (001) and the slip direction is toward the dodecahedron face (110). In the case of cubic thallium halide crystals the slip plane is parallel to the dodecahedron face (110) and the slip direction is toward the cube face (001). This difference in behavior may be caused by the different lattice structures. Both crystals are cubic, but while the lattice of sodium chloride is face centered, the lattice of cubic thallium halides is body centered. In both cases the slip planes contain positive and negative ions. The slip direction is in both cases toward planes which contain only negative or positive ions.

10. Explanation of Punch Patterns. On the bases of the mechanism of plastic deformation previously discussed, it is possible to explain the change of patterns with respect to the crystal orientation. It is necessary to consider two points: the slip planes and the slip directions. By pressing a cone into a crystal surface press forces are produced in radial directions from the press point. Only the forces which are parallel to the cube planes (slip directions) cause the plastic deformations which result in superficial and transmitted patterns. Press forces in other directions produce strains. The possible deformation patterns, their shape and position for three principal orientations are indicated in Figs. 19, 20, and 21. Pressing on a cube face produces four symmetrical superficial wings and one transmitted pattern; on the dodecahedron face two superfical wings and two transmitted patterns; and on the octahedron face three superfical wings and three transmitted patterns.

11. Use of Superficial Patterns for Determination of Crystal Orientation. Superficial patterns permit a quick and simple determination of crystal orientation within a few degrees. It is not

9. A. F. Joffe', The Physics of Crystals, (New York: McGraw Hill, 1928).

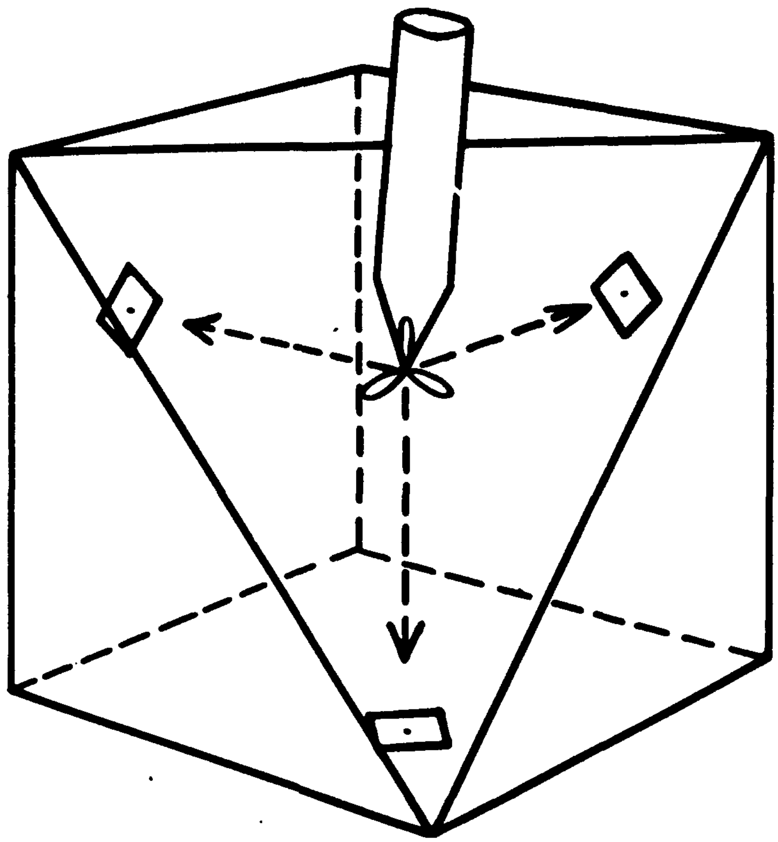


Fig. 21. Superficial and transmitted patterns formed by pressing on an octahedron face.

necessary to cut, grind, or polish the crystal. The crystal can be used in its form after growing. A cone is pressed by hand into the crystal surface (a force of about 10 pounds is sufficient) and the resulting pattern is compared with patterns in Fig. 24. This comparison permits rough orientation and more accurate orientation is achieved by additional punching until the cube (100), dodecahedron (110), or octahedron (111) face, is found. The patterns show fourfold symmetry for the (100), twofold for the (110), and threefold for the (111) face, as can be seen in Figs. 24. Since the patterns change rapidly with small deviations from the above-mentioned faces, the orientation of the crystal may be accurately determined.

Orientation of the crystal must be known in order to determine properly its mechanical properties. These properties as well as the optical properties are important to the instrument designer. Knowledge of the crystal orientation is also valuable in the processing of the crystal into optical elements. Polishing on the cube face, for instance, results in the best polish in the shortest time.

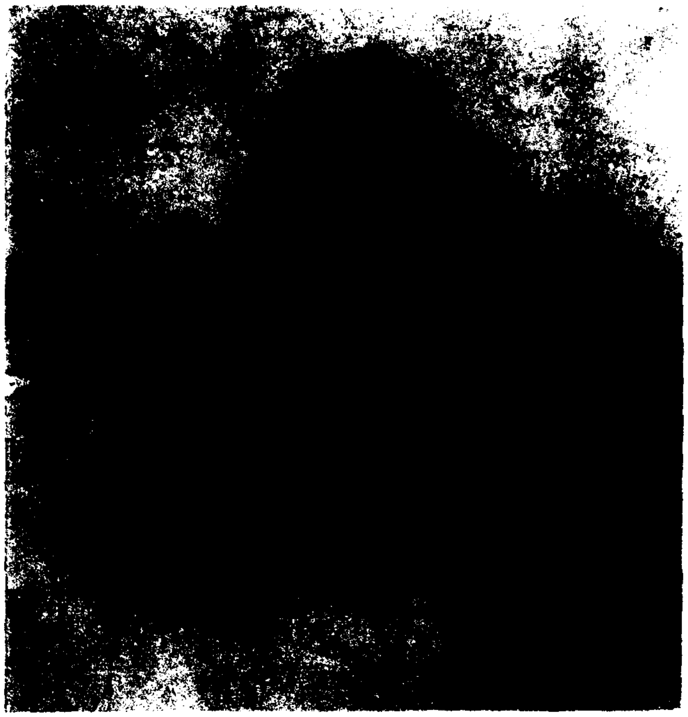


Fig. 22. Press pattern on silver chloride crystal.

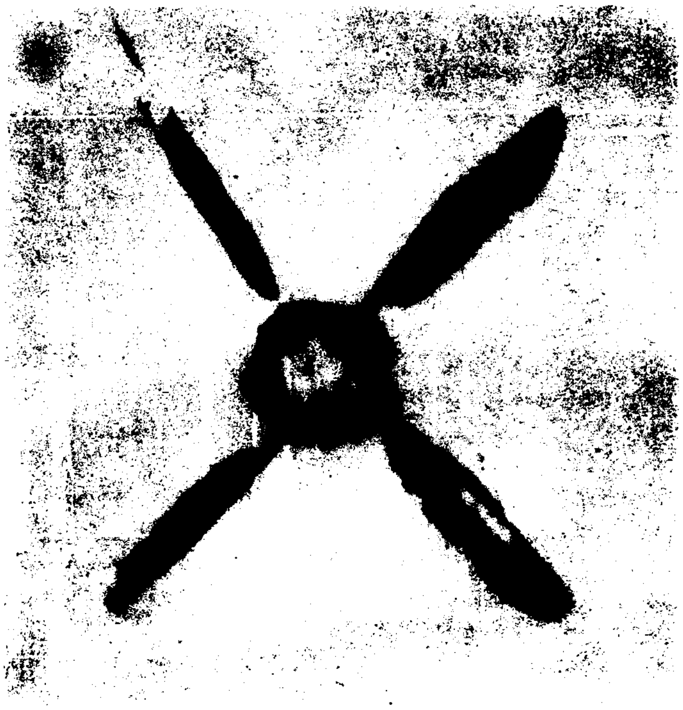


Fig. 23. Press pattern on cube face of NaCl.

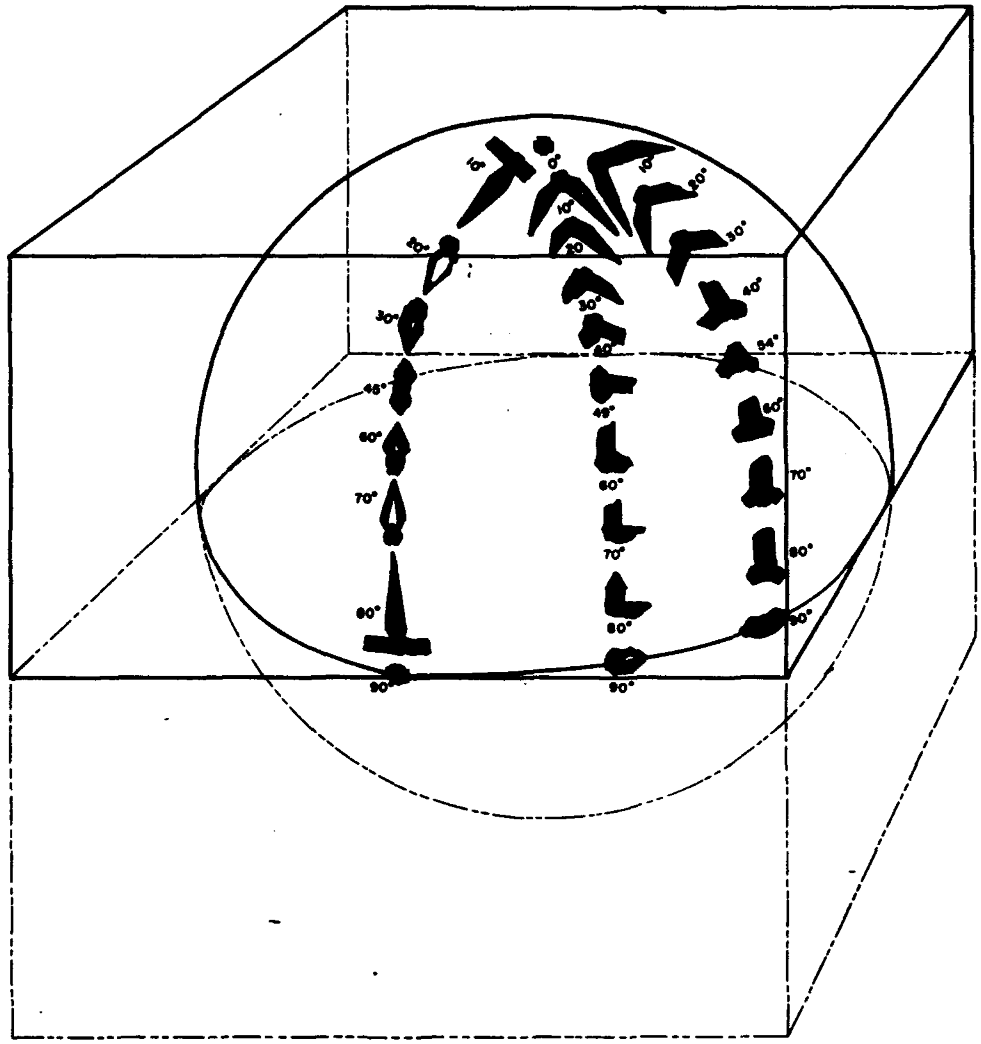


Fig. 24. Orientation of KRS-5 and KRS-6 as determined by press patterns.

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HALIDES IN RELATION TO CRYSTAL

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400.1 (8-23-02-002)

SUBJECT: Transmittal of Report 1102, The Plastic Deformation of Thallium Halides in Relation to Crystal Orientation

THRU: Commanding General
The Engineer Center and Fort Belvoir
Fort Belvoir, Virginia

TO: Chief of Engineers
Department of the Army
ATTENTION: Chief, Engineer Research and Development Division

1. Transmitted herewith is Report 1102, "The Plastic Deformation of Thallium Halides in Relation to Crystal Orientation," dated 14 January 1949, which was prepared by the Technical Staff of the Engineer Research and Development Laboratories. This report covers an investigation of the plastic deformation of cubic crystals of thallium halides in relation to crystal orientation.

2. The plastic deformations of cubic thallium halide crystals, produced under pressure of a cone perpendicular to the crystal surface, are seen as two kinds of surface patterns, the superficial and the transmitted, with characteristics dependent on the crystal orientation. Superficial patterns have wings radiating from the press point; transmitted patterns are square or rhombic. The number, orientation, shape, and length of the wings of the superficial patterns, and the shape and position of the transmitted patterns, depend on the orientation of the crystal with respect to the surface of the crystal samples. Characteristics of the patterns are explained by the mechanism of plastic deformation in crystals. Application of the punch patterns to determination of crystal orientation preparatory to processing the crystals into optical elements is explained.

3. The report and its contents is approved.

2 Incls
1. Proposed Distr List
2. Rpt as above (in dup)

John C. Arrowsmith
JOHN C. ARROWSMITH
Colonel, CE
Commanding

96

BASIC: Ltr. dtd 16 Feb 1948, subj: Trans. of Report 1102, The Plastic Deformation of Thallium Halides in relation to Crystal Orientation. ERDL.

TECAG 1st Ind

Headquarters, The Engineer Center & Ft. Belvoir, Fort Belvoir, Virginia.

TO: Chief of Engineers, Department of the Army, Washington, D. C.
ATTENTION: Chief, Engineer Research and Development Division

2 Incls.
n/c

SUBJECT: Transmittal of Report 1102, The Plastic Deformation of Thallium Halides in Relation to Crystal Orientation

ENGNA (16 Feb 49) 2nd Ind.

Office of the Chief of Engineers, Washington 25, D. C. 28 February 1949

To: The Commanding General, The Engineer Center, Fort Belvoir, Virginia

The Report including the proposed distribution list is approved.

BY ORDER OF THE CHIEF OF ENGINEERS:

H. J. WOODBURY
Colonel, Corps of Engineers
Chief, Engr. Research & Development Div.
Military Operations

1 Incl.
1 and 2 w/d
Added:
3. Cpy ltr to Dir.Log.
GSUSA dtd 28 Feb 49.

TECAG 3d Ind

Headquarters, The Engineer Center & Fort Belvoir, Fort Belvoir, Virginia

TO: Commanding Officer, Engineer Research & Development Laboratories, Fort Belvoir, Virginia

1 Incl
n/c

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